PATENT SPECIFICATION 724,751

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COMPLETE SPECIFICATION.

Improvements in the Manufacture of Titanium Pigments.

We, LAPORTE TITANIUM LIMITED, formerly known as NATIONAL TITANIUM PRODUCTS LIMITED, a British Company, of Kingsway, Luton, Bedfordshire, JAMES THOMSON RICHMOND, a British Subject, of 230 Stockingstone Road, Luton, Bedfordshire, and JAMES TAYLOR, a British Subject, of 66 Oakley Road, Luton, Bedfordshire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement :—

Various processes have been proposed for improving the properties of titanium pigments by depositing water-insoluble metal compounds on the particles of the pigments in aqueous suspension. United States Specification No. 1,368,392 describes adding to such an aqueous suspension first a solution of a salt or salts of a metal or metals, referred to as aluminium, calcium, lead, zinc and similar metals, and then a precipitant, for example, sodium carbonate or hydroxide, is introduced to precipitate a white and relatively insoluble metal compound or compounds upon the particles of the pigment. The treatment is said to stabilise the pigment against chemical and physical action, and especially to prevent discoloration of the paint in which the pigment is used. United States Specification No. 2,378,790 describes preparing an aqueous slurry of a titanium pigment, if desired, with the aid of a dispersing agent, and precipitating *in situ* upon the suspended particles of the pigment a white insoluble silicate of a metal of the second, third and fourth groups of the periodic system, for example, magnesium, zinc, aluminium, yttrium, zirconium or cerium. The treatment is said to improve the resistance to chalking, gloss retention and resistance to discoloration of coating compositions in which the treated pigments are incorporated.

[Price 3/-d.]

The present invention provides a process for the treatment of titanium pigments which leads to an improvement in their tinting strength, opacity, and capacity for being wetted by and dispersed in organic paint media, and to improved flow characteristics of paints in which the pigments are incorporated. The term "titanium pigments" is used herein to denote calcined titanium dioxide pigments and composite pigments containing a calcined titanium dioxide pigment.

According to this invention a process for improving a titanium pigment, comprises dispersing the pigment in an aqueous medium by the action of a dispersing agent and agitation, adding to the dispersion without flocculating it a water-soluble magnesium salt and a water-soluble aluminium salt each in a proportion corresponding to 0.1 to 2.0 per cent of MgO and Al_2O_3, respectively, calculated on the weight of the pigment, and then precipitating the magnesium and aluminium in the form of water-insoluble compounds on the pigment particles by means of a compound of alkaline reaction.

Any desired dispersing agent may be used for dispersing the titanium pigment, and as examples there may be mentioned sodium hexametaphosphate, trisodium phosphate or sodium hydroxide. The pigment may be dispersed in water at any convenient temperature with the aid of, for example, 0.1 to 2.0 per cent of the dispersing agent calculated on the weight of the titanium pigment.

The water-soluble salts of magnesium and aluminium may, for example, be magnesium sulphate and aluminium sulphate. The aluminium addition leads to an improvement in the wettability of the pigment in paint media and in the flow characteristics of paint containing the pigment. The magnesium addition, on the other hand, enhances the tinting strength and opacity of the pigment,

Price 4s 6d. Price 25p

and tends, if present in a high proportion, slightly to diminish the flow characteristics. This can, however, be counteracted by increasing the proportion of the aluminium addition. Generally speaking, within the above range of 0.1 to 2.0 per cent, a high proportion of alumina relatively to magnesia will favour the wettability and flow characteristics and a high proportion of magnesia relatively to alumina will favour the tinting strength and opacity.

The ratio of the dispersing agent to the magnesium salt (calculated as MgO) advantageously ranges from 1 : 0.5 to 1 : 2, and the ratio of the dispersing agent to the combined magnesium and aluminium salt additions (calculated as $MgO + Al_2O_3$) preferably ranges from 1 : 1 to 1 : 5.

In order still further to improve the wettability and flow characteristics, without diminishing the tinting strength of the pigment, a water-soluble titanium salt may also be added to the dispersion in a proportion corresponding to not more than 2 per cent of TiO_2, calculated on the weight of the pigment, the titanium then being precipitated in the form of a water-insoluble compound together with the magnesium and aluminium compounds upon the addition of the compound of alkaline reaction.

After the addition of the water-soluble salts, a compound of alkaline reaction, for example, sodium hydroxide or sodium carbonate, is added to precipitate the magnesium and aluminium, and also the titanium, if present, in the form of water-insoluble compounds. This will be brought about by adjusting the *p*H of the dispersion by means of the alkaline compound to the value required to effect the precipitation. The *p*H value will generally be adjusted to within the range of 6.5 to 8.5.

During the addition and precipitation of the metal salts the dispersion should be vigorously agitated. The treated pigment may then be washed to remove water-soluble compounds, and separated by filtration and dried. Alternatively, the treated pigment, without being dried, may be flushed with oil to form an oil paste.

The following Examples illustrate the invention :—

EXAMPLE 1.

1000 pounds of ground calcined titanium dioxide are slurried in 400 gallons of water containing 10 pounds of sodium hexametaphosphate. The slurry is agitated to ensure uniform distribution and dispersion of the pigment. To the resulting dispersion is added with stirring a solution of magnesium sulphate containing the equivalent of 6 pounds of MgO in 15 gallons of water. This is followed by the addition of a solution of aluminium sulphate containing the equivalent of 4 pounds of Al_2O_3 in 10 gallons of water. During the additions the dispersion is thoroughly agitated. The *p*H value of the dispersion is then adjusted to within the range of 7.0 to 7.5 by the addition of a solution of sodium hydroxide. The treated pigment is then washed with water to remove sodium sulphate, and is separated by filtration, dried and disintegrated.

A calcined titanium dioxide pigment having a tinting strength of 1550 on the Reynold's scale had this value raised to 1760 by the treatment described in this Example. 150 parts of a pigment so treated, when tested under standard conditions in admixture with 80 parts of oil, gave a flow of 8.4 centimetres as compared with 2.6 centimetres for a mixture of the untreated pigment and oil in the same proportions.

EXAMPLE 2.

1000 pounds of ground calcined titanium dioxide are slurried in 400 gallons of water containing 5 pounds of sodium hexametaphosphate. The slurry is agitated to ensure uniform distribution and dispersion of the pigment. To the dispersion is added with stirring a solution of magnesium sulphate containing the equivalent of 3 pounds of MgO in 7.5 gallons of water. This is followed by the addition of a solution of titanyl sulphate containing the equivalent of 3 pounds of TiO_2 in 15 gallons of water and a solution of aluminium sulphate containing the equivalent of 3 pounds of Al_2O_3 in 7.5 gallons of water. During the additions, the dispersion is thoroughly agitated. The *p*H value of the dispersion is then adjusted to within the range of 7.0 to 7.5 by the addition of a solution of sodium hydroxide. The treated pigment is then washed with water to remove sodium sulphate, and is separated by filtration, dried and disintegrated.

A calcined titanium dioxide pigment having a tinting strength of 1500 on the Reynold's scale had this value raised to 1700 by the treatment described in this Example. 150 parts of a pigment so treated, when tested under standard conditions in admixture with 80 parts of oil, gave a flow of 20.8 centimetres compared with 2.6 centimetres for a mixture of the untreated pigment and oil in the same proportions.

What we claim is :—

1. A process for improving titanium pigments, wherein a titanium pigment as hereinbefore defined is dispersed in an aqueous medium by the action of a dispersing agent and agitation, a water-soluble magnesium salt and a water-soluble aluminium salt each in a proportion corresponding to 0.1 to 2.0 per cent of MgO and Al_2O_3, respectively, calculated on the weight of the pigment, are added to the dispersion without causing the latter to flocculate, and the magnesium and

aluminium are precipitated in the form of water-insoluble compounds on the pigment particles by means of a compound of alkaline reaction.

2. A process as claimed in Claim 1, wherein magnesium sulphate and aluminium sulphate are used as the magnesium and aluminium salts.

3. A process as claimed in Claim 1 or 2, wherein sodium hexa-metaphosphate, trisodium phosphate or sodium hydroxide is used as dispersing agent.

4. A process as claimed in Claim 1, 2 or 3, wherein sodium hydroxide or sodium carbonate is used as the compound of alkaline reaction.

5. A process as claimed in any one of Claims 1—4, wherein the ratio of the dispersing agent to the magnesium salt (calculated as MgO ranges from 1 : 0.5 to 1 : 2.

6. A process as claimed in any one of Claims 1—5, wherein the ratio of the dispersing agent to the combined magnesium and aluminium salt additions (calculated as MgO and Al_2O_3) ranges from 1 : 1 to 1 : 5.

7. A process as claimed in any one of Claims 1—6, wherein a water-soluble titanium salt is also added to the dispersion in a proportion corresponding to not more than 2 per cent of TiO_2 calculated on the weight of the pigment, and the titanium is precipitated in the form of a water-insoluble compound together with the magnesium and aluminium compounds by means of the compound of alkaline reaction.

8. A process for improving a titanium pigment conducted substantially as described in Example 1 or Example 2 herein.

9. Titanium pigments which have been improved by the process claimed in any one of Claims 1—8.

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PROVISIONAL SPECIFICATION.

Improvements in the Manufacture of Titanium Pigments.

We, NATIONAL TITANIUM PIGMENTS LIMITED, a British Company, of Kingsway, Luton, Bedfordshire, JAMES THOMSON RICHMOND, a British Subject, of 230 Stockingstone Road, Luton, Bedfordshire, and JAMES TAYLOR, a British Subject, of 66 Oakley Road, Luton, Bedfordshire, do hereby declare the nature of this invention to be as follows :—

Various processes have been proposed for improving the properties of titanium pigments after calcination, in which a water-insoluble aluminium compound is deposited on the particles of an aqueous suspension of the pigment, for example, by adding a water-soluble aluminium salt to the suspension and precipitating the aluminium on the pigment particles in the form of a water-insoluble aluminium compound by means of a water-soluble alkali hydroxide or silicate, or by adding the aluminium in the form of a basic aluminium salt. These processes are said to improve such properties of the pigments as their resistance to chalking, tint retention, gloss retention and after-yellowing.

The present invention provides a process for the treatment of titanium pigments which leads to an improvement in their tinting strength, opacity, and capacity for being wetted by and dispersed in organic paint media, and to improved flow characteristics of paints in which the pigments are incorporated. The term "titanium pigments" is used herein to denote calcined titanium dioxide pigments and composite pigments containing a calcined titanium dioxide pigment.

According to this invention a process for improving a titanium pigment, comprises dispersing the pigment in an aqueous medium by the action of a dispersing agent and agitation, adding a small proportion of a water-soluble magnesium salt and a water-soluble aluminium salt to the dispersion without flocculating it, and then precipitating the magnesium and aluminium in the form of water-insoluble compounds on the pigment particles by means of a compound of alkaline reaction.

Any desired dispersing agent may be used for dispersing the titanium pigment, and as examples there may be mentioned sodium hexametaphosphate, trisodium phosphate or sodium hydroxide. The pigment may be dispersed in water at any convenient temperature with the aid of, for example, 0.1 to 2.0 per cent of the dispersing agent calculated on the weight of the titanium pigment.

The water-soluble salts of magnesium and aluminium may, for example, be magnesium sulphate and aluminium sulphate. The proportions of the magnesium and aluminium salts added will generally not exceed amounts corresponding to 2 per cent of MgO and Al_2O_3, respectively, calculated on the weight of the pigment. A suitable range in each case is 0.1 to 2.0 per cent. The aluminium addition leads to an improvement in the wettability of the pigment in paint media and in the flow characteristics of paint containing the pigment. The magnesium addition, on the other hand, enhances the tinting strength and opacity of the pigment, and tends, if

present in a high proportion, slightly to diminish the flow characteristics. This can, however, be counteracted by increasing the proportion of the aluminium addition. Generally speaking, within the above range of 0.1 to 2.0 per cent, a high proportion of alumina relatively to magnesia will favour the wettability and flow characteristics and a high proportion of magnesia relatively to alumina will favour the tinting strength and opacity.

The ratio of the dispersing agent to the magnesium salt (calculated as MgO) advantageously ranges from 1 : 0.5 to 1 : 2, and the ratio of the dispersing agent to the combined magnesium and aluminium salt additions (calculated as $MgO + Al_2O_3$) preferably ranges from 1 : 1 to 1 : 5.

In order still further to improve the wettability and flow characteristics, without diminishing the tinting strength of the pigment, a small proportion of a water-soluble titanium salt may also be added to the dispersion, the titanium then being precipitated in the form of a water-insoluble compound together with the magnesium and aluminium compounds upon the addition of the compound of alkaline reaction. The proportion of the titanium salt added will generally not exceed an amount corresponding to 2 per cent of TiO_2 calculated on the weight of the pigment.

After the addition of the water-soluble salts, a compound of alkaline reaction, for example, sodium hydroxide or sodium carbonate, is added to precipitate the magnesium and aluminium, and also the titanium, if present, in the form of water-insoluble compounds. This will be brought about by adjusting the *p*H of the dispersion by means of the alkaline compound to the value required to effect the precipitation. The *p*H value will generally be adjusted to within the range of 6.5 to 8.5.

During the addition and precipitation of the metal salts the dispersion should be vigorously agitated. The treated pigment may then be washed to remove water-soluble compounds, and separated by filtration and dried. Alternatively, the treated pigment, without being dried, may be flushed with oil to form an oil paste.

The following Examples illustrate the invention :—

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A calcined titanium dioxide pigment having a tinting strength of 1550 on the Reynold's scale had this value raised to 1760 by the treatment described in this Example. 150 parts of a pigment so treated, when tested under standard conditions in admixture with 30 parts of oil, gave a flow of 8.4 centimetres as compared with 2.6 centimetres for a mixture of the untreated pigment and oil in the same proportions.

EXAMPLE 2.

1000 pounds of ground calcined titanium dioxide are slurried in 400 gallons of water containing 5 pounds of sodium hexametaphosphate. The slurry is agitated to ensure uniform distribution and dispersion of the pigment. To the dispersion is added with stirring a solution of magnesium sulphate containing the equivalent of 3 pounds of MgO in 7.5 gallons of water. This is followed by the addition of a solution of titanyl sulphate containing the equivalent of 3 pounds of TiO_2 in 15 gallons of water and a solution of aluminium sulphate containing the equivalent of 3 pounds of Al_2O_3 in 7.5 gallons of water. During the additions, the dispersion is thoroughly agitated. The *p*H value of the dispersion is then adjusted to within the range of 7.0 to 7.5 by the addition of a solution of sodium hydroxide. The treated pigment is then washed with water to remove sodium sulphate, and is separated by filtration, dried and disintegrated.

A calcined titanium dioxide pigment having a tinting strength of 1500 on the Reynold's scale had this value raised to 1700 by the treatment described in this Example. 150 parts of a pigment so treated, when tested under standard conditions in admixture with 30 parts of oil, gave a flow of 20.8 centimetres compared with 2.6 centimetres for a mixture of the untreated pigment and oil in the same proportions.

Dated this 26th day of April, 1949.

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